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METHOD OF MAKING INTERMETALLIC COMPOUND-COMPOSITION BODIES

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This invention relates to binary intermetallic compound-compositions, to bodies composed thereof, and to methods of making the compound-compositions and bodies thereof.

The invention relates more specifically to binary intermetallic compound-composition bodies which are of fine grain, having substantially the theoretical density of the intermetallic compound-composition of which they are composed, and which, at temperatures ranging from as high as 2000° F. to 2900° F., have extremely high resistance to oxidation and extremely great strength.

Important features reside in the steps of the preferred method by which the starting binary intermetallic compound-compositions in the powdered form are produced, and of the preferred method by which the powders are formed into the bodies having the physical properties above described.

Another feature is to form the bodies of the basic binary intermetallic compound-compositions in which the metals are of such non-stoichiometric proportions as to increase the mechanical strength at the elevated temperatures over that which the bodies would have if the proportions of the two metals were stoichiometric.

Various objects and advantages of the invention will become apparent from the following description wherein specific examples of the compound-compositions and methods of producing the same and the bodies are shown by way of illustration.

The binary intermetallic compound-compositions of the present invention, in some cases, partake of the characteristics of compounds, and in other cases, of the characteristics of alloys. In some cases their exact nature is not determinable with certainty. Accordingly, they are all referred to hereinafter as "compound-compositions."

The binary intermetallic compound-compositions of the present invention consist essentially of two metals, one selected from a first group consisting of beryllium and aluminum, and another metal M selected from a second group consisting of niobium, zirconium, tantalum and molybdenum.

By the term "consisting essentially of," it is meant to exclude any other metal or materials subversive of the characteristics of the substantially pure binary intermetallic compound-compositions.

The beryllium from the first group can be combined with any one of the metals from the second group. The aluminum of the first group can be combined with any one of the metals of the second group, but the outstanding advantages of the binary intermetallic compound-compositions containing aluminum are obtainable only when the aluminum is combined with either niobium or tantalum.

The ratio, by weight, of the metal of the first group to that of the second group can be any ratio desired in a broad range wherein metal from neither group is less than about 20% of the combined weight of the two

metals, the balance consisting essentially of a metal from the second group, and the beryllium from the first group does not exceed substantially its stoichiometric proportion corresponding to MBe_{12} and MBe_{13} and the aluminum from the first group does not exceed substantially its stoichiometric proportion corresponding to MAl_3, wherein M is a metal selected from the group consisting of niobium, zirconium, tantalum and molybdenum. Some of the metals, when used within more limited ratios within these broader ranges, have additional and particularly outstanding qualities.

The metals of both groups preferably are substantially pure. For the most outstanding results, the binary intermetallic compound-compositions hereof preferably have total impurities not exceeding about 1%, by weight, of their total weight.

The methods of producing the basic intermetallic compound-compositions are as follows:

In one method, the selected metals, one from each group, in the form of powder of particle size of about 50 mesh or finer, are intimately intermixed and reacted to completion. They may be reacted by melting in which case the resultant product is extremely hard and difficult to pulverize to form a powder for forming the final bodies.

The preferred methods are to effect the reaction of the metals in a solid state, at sintering temperatures, and below the temperature at which appreciable melting or fusion would occur, to produce a friable compact of the binary intermetallic compound-compositions, which compact is readily pulverizable to a particle size of about 200 mesh or finer. In the latter method, the two intimately intermixed metallic powders are first subjected to high mechanical pressure at room temperature to form a compact.

The compact is then charged into a graphite crucible, having a molybdenum liner, wherein the compact rests in the crucible, on a beryllium oxide plate. The crucible mouth is covered with a beryllium oxide plate. The crucible is then subjected to vacuum in a furnace and the pressure in the furnace is thereby lowered, preferably to less than one micron of mercury.

Heat is then applied, starting at room temperature, preferably by means of an adjustable electric resistance heating element so that accurate control of the heating is obtainable. The heat is applied so as gradually to bring the compact up to a temperautre of about 450° C. During this initial heating period, moisture and occluded and evolved gases are driven out of the compact and drawn off by the vacuum pump from the crucible and compact. As they are evolved and freed, the furnace pressure tends to rise, but is maintained generally at less than ten microns, and preferably at less than one micron of mercury, by the continuance of the evacuation.

As one example of a compact, about 170 cubic centimeters, by volume, of the mixture of the two metallic powders are subjected preferably to about 30 tons per square inch of mechanical pressure, at room temperature. This produces a cold compact of about 65% of theoretical density.

As an alternate method, the compacting step may be omitted and the selected mixture of powdered metals may be reacted in loose form, the other steps, conditions, and proportions, remaining unchanged.

Compacting has the advantage of convenience in handling the metals and charging the crucible. In addition, there is less danger of contamination of the charge

by reaction with the container due to minimum surface contact in the case of the compact.

In some cases, particularly when the starting metals have not been pre-dried and de-gassed, heating is continued for as much as 16 hours, and during the entire period, the vacuum is maintained. In either event, after the initial heating to about 450° C., the temperature is increased at the rate of about 100° C. per hour until it reaches a higher temperature of from about 1000° C. to 1400° C. This higher temperature, together with the vacuum, is continued for about one hour.

Next the heat is turned off, and with the vacuum continued, the compact is allowed to cool to less than 100° C., after which it is removed from the furnace.

The exact period of time of heating may be varied considerably, the controlling limitation, however, being that if the heating is too rapid, the metallic powders melt or fuse to an extent which renders the resultant mass solid and unfriable.

When the gradual heating step is employed, the resultant compact is very porous and friable and can be pulverized readily to a particle size of 200 mesh or finer to provide the initial powder for practicing the process of forming the bodies, as hereinafter described.

By starting with the recited initial heating time of 16 hours, and gradually reducing it for each run, the optimum time for processing each mixture can be established without loss of valuable metal.

In the foregoing sintering methods, an inert atmosphere may be substiuted for the vacuum throughout the practice of the entire method. However, to obtain compositions of the highest purity, the mixture should be substantially evacuated prior to reaction in the inert atmosphere.

The following Examples 1–9 are illustrative of the preparation of the basic powder of intermetallic compound-compositions, and are specific to our method whether the compacting step is omitted or not, and whether or not the recited substitution of inert atmosphere for vacuum is made, the other steps, conditions, and proportions remaining unchanged.

EXAMPLE 1.—BERYLLIUM-NIOBIUM

Finely powdered beryllium, in an amount of 3.50 kilograms, was intimately mixed with 3.017 kilograms of the finely powdered niobium, both materials being of a particle size of 50 mesh or finer. The intimate intermixture was then compacted at room temperature at a pressure of about 30 tons per square inch. The cold pressed compact was placed in the graphite crucible in a furnace chamber which was then evacuated while at room temperature to a pressure of less than one micron of mercury. As soon as this degree of evacuation was reached, heat was applied and the compact heated gradually to 450° C., while continuing the evacuation. The heating and evacuation were continued until the compact was substantially free from moisture and occluded gases and vapors. Thereupon the rate of heating was increased by about 100° C. per hour until a maximum temperature of about 1270° C. was reached, the vacuum meanwhile being continued. This maximum temperature and concurrent evacuation were continued for about one hour. Thereupon the heating was then discontinued, but with the vacuum continuing, the furnace was cooled to room temperature. The product of this reaction was a friable and porous compact. It was removed from the furnace and was easily pulverized to powder having a particle size of 200 mesh or finer. The beryllium content determined by chemical analysis was 53.6% by weight, of the intermetallic compound-composition, the balance consisting essentially of niobium.

EXAMPLE 2.—BERYLLIUM-NIOBIUM

The procedure of Example 1 was followed using 535.4 grams of beryllium and 648.6 grams of niobium. The beryllium content, found by chemical analysis, was 45.2% by weight of the intermetallic compound-composition, the balance consisting essentially of niobium.

EXAMPLE 3.—BERYLLIUM-ZIRCONIUM

The procedure of Example 1 was followed using 3.461 kilograms of beryllium and 2.644 kilograms of zirconium. By chemical analysis, the beryllium content was 56% by weight of the intermetallic compound-composition, the balance consisting essentially of zirconium.

EXAMPLE 4.—BERYLLIUM-ZIRCONIUM

The procedure of Example 1 was followed using 524.3 grams of beryllium and 622.7 grams of zirconium. The beryllium content determined by chemical analysis was 45.7% by weight of the intermetallic compound-composition, the balance consisting essentially of zirconium.

EXAMPLE 5.—BERYLLIUM-TANTALUM

The procedure of Example 1 was followed using 1.908 kilogrmas of beryllium and 3.192 kilograms of tantalum. The beryllium content, determined by chemical analysis, was 37.2% by weight of the intermetallic compound-composition, the balance consisting essentially of tantalum.

EXAMPLE 6.—BERYLLIUM-TANTALUM

The procedure of Example 1 was followed using 540.2 grams of beryllium and 1247 grams of tantalum. The beryllium content, determined by chemical analysis, was 29.8% by weight of the intermetallic compound-composition, the balance consisting essentially of tantalum.

EXAMPLE 7.—BERYLLIUM-MOLYBDENUM

The procedure of Example 1 was followed using 2.676 kilograms of beryllium and 2.354 kilograms of molybdenum. By chemical analysis, the beryllium content was 53% by weight of the intermetallic compound-composition, the balance consisting essentially of molybdenum.

EXAMPLE 8.—ALUMINUM-NIOBIUM

The procedure of Example 1 was followed using 282 grams of aluminum and 324 grams of niobium. The aluminum content, determined by chemical analysis, was 46.5% by weight of the intermetallic compound-composition, the balance consisting essentially of niobium.

EXAMPLE 9.—ALUMINUM-TANTALUM

The procedure of Example 1 was followed using 258 grams of aluminum and 577 grams of tantalum. By chemical analysis, the aluminum content was 30.9% by weight of the intermetallic compound-composition, the balance consisting essentially of tantalum.

Having prepared the basic pulverized intermetallic compound-composition powder, the next step is to form any selected one of them into bodies having a fine grain, a density of substantially the calculated theoretical density, and high oxidation resistance and high strength at temperatures of from 2000–2900° F. To this end, the pulverized intermetallic compound-composition powder, preferably of a particle size of 200 mesh or finer, is charged into a graphite die having substantially the internal configuration desired for the exterior of the body to be produced.

The charge and die are subjected, in a furnace at room temperature, to concurrent evacuation and application of mechanical pressure of about 1000 pounds per square inch. The evacuation and mechanical pressure are continued until the furnace chamber, with the charged die therein, is evacuated to a furnace pressure not to exceed 1000 microns of mercury, and preferably as low as 40 microns of mercury. Beginning when the furnace pressure has reached relative stability, indicating that substantially all the gases and vapors have been removed, and with the mechanical pressure and the vacuum continued, heat is applied.

The temperature is increased and the mechanical pressure is concurrently increased to a maximum of about 2000 pounds per square inch as the temperature increases until substantially complete compaction and maximum density are obtained, usually at about 1400° C. to about 1650° C. When this latter temperature and pressure are reached, the application of mechanical pressure is discontinued, but the vacuum and elevated temperature are continued thereafter until the stresses developed in the body of the preceding steps are relieved. This latter step, which, in general, is an annealing step, generally requires about one-half hour for small compacts.

After the body is annealed, heating is discontinued and the furnace, die, and body are cooled gradually, under vacuum, to room temperature, after which the body is ready for removal and use.

As mentioned, the beryllium and aluminum may be used in amounts as low as 20%, by weight of the body, up to but not to exceed stoichiometric proportions of, in the case of beryllium, MBe_{12} and MBe_{13}, and in the case of aluminum, MAl_3, wherein M is a metal selected from the group consisting of niobium, zirconium, tantalum and molybdenum, in any instance. If either beryllium or aluminum is used in amounts less than stoichiometric proportions, it cannot with certainty be said that the resultant material is a true compound. Instead, it partakes somewhat of the nature both of a true compound and a mixture of compounds.

A large number of intermetallic compound-compositions, some of which are of various phases, can be produced by selecting predetermined ratios of the selected starting powders.

The strength and oxidation resistance properties of the various intermetallic compound-compositions, at the elevated temperatures, vary for different proportions of the selected metals.

Generally, when a greater proportion of beryllium or aluminum than a stoichiometric proportion of the more beryllium-rich, MBe_{12} and MBe_{13}, and aluminum-rich, MAl_3, respectively, compound-compositions of our invention is used as the upper limit of the range, in producing the binary intermetallic compound-composition powders, the compound-composition loses its high strength and oxidation resistance properties at the elevated temperatures. The loss of these desirable properties is believed to be attributable to the presence of free or unreacted beryllium or aluminum, either of which melts at a lower temperature than 2350° F. In the case of very small amounts of the free metal, porosity and cracking result, and in the case of larger amounts, the melting prevents the formation of a solid and dense body, at the hot-pressing temperatures employed in our method. These upper substantially stoichiometric limits of beryllium or aluminum of the above beryllium-rich and aluminum-rich compound-compositions of our invention are the upper critical limits, and preferably should not be exceeded at all, and certainly by not more than a small fraction of a percent. Surprisingly, the inclusion of these metals in less than stoichiometric proportions produces, in some instances, the greatest mechanical strength at elevated temperatures.

On the other hand, the lower limits of the specified ranges of beryllium and aluminum are not as critical, since the intermetallic compound-compositions containing ten to fifteen percent, by weight, of beryllium or aluminum are nevertheless of high melting point. The recited ranges, however, cover those intermetallic compound-compositions which exhibit the most favorable grain size, density, oxidation resistance, and high strength, at elevated temperatures from 2000–2900° F.

It should be realized that the metal powders employed in the preparation of the intermetallic compound-compositions, as in the case of most metals, contained minor impurities. However, the beryllium powder used was 99.0% or more pure, and contained mainly beryllium oxide and very minor amounts of heavy metals and aluminum as impurities. Such high purity is preferred, but impurities up to about 3% may be tolerated in some instances. The niobium, tantalum, molybdenum, and zirconium powders used were at least about 99.5% pure.

Analysis of the examples hereinafter given discloses that the binary intermetallic compound-compositions of the present invention were not 100%, or pure, but typically contained one or more of the following impurities in the percentages indicated.

Impurity:	Percent by weight of binary intermetallic compound-composition
O_2	0.8–1.0
Fe	0.05–0.07
Al	0.01–0.02
Si	0.001–0.01
C	0.07–0.09
Cr, Ni, Mg, Mn (total)	0.010–0.015

The strength and oxidation resistance properties of bodies of a large number of the binary intermetallic compound-compositions of the present invention were evaluated by well known "modulus-of-rupture" and "gain-in-weight" oxidation testing procedures. These and other results are presented in Tables I and II included hereinbelow. Said tables are referenced in some instances to the specific examples included in this specification. The gain-in-weight data were obtained by exposing weighed specimen bodies of each intermetallic compound-composition to a 100 cubic centimeter per minute flow of either dry or moist air for 100 hours at the respective temperatures indicated in Table II hereof. The specimens were then re-weighed to determine the increase in weight which is representative of the oxidation resistance for each composition.

The penetration data, also indicative of the oxidation resistance of these compositions, were calculated from the gain-in-weight data, on the assumption that the gains occurred by oxidation and that all compositions oxidized stoichiometrically. The compound-compositions which showed penetration of less than 2 mils in 100 hour exposure are considered to have good oxidation resistance in the indicated temperature ranges.

Examples of the method of forming the bodies, and physical properties of the formed bodies follow. Some of the relevant data are tabulated and referenced in Tables I and II by reference to their example numbers.

Examples of the formation of bodies by the foregoing method are as follows:

EXAMPLE 10

(Specimen A of tables)

An intermetallic compound-composition powder, in an amount of 176.4 grams, consisting of 53.6%, by weight, of beryllium and the balance consisting essentially of niobium, and having a particle size of 200 mesh or finer, was placed in a furnace in a graphite die and mechanical pressure at about 1000 pounds per square inch was then applied to the powder composition and then initially evacuated while at room temperature. Beginning when the gases and vapors were substantially evacuated, heat was applied while maintaining mechanical pressure and vacuum. The temperature was increased gradually and the mechanical pressure was concurrently increased, the vacuum concurrently being continued, until a temperature of about 1550° C. and a pressure of about 2000 p.s.i. were reached. The mechanical pressure was then discontinued, the vacuum and temperature being continued for about another 20 minutes. Thereupon, the heating was discontinued, and the furnace was allowed to cool, under vacuum, to room temperature. The body, upon removal from the furnace, weighed 167.6 grams and had a density of 2.88 g./cc., which is 98% of the calculated theoretical density. The grain size was 11 microns, and the beryllium content, determined by chemical analysis, was

53.6%, by weight, of the body, the balance consisting essentially of niobium.

The furnace pressure reached during the initial substantial evacuation of the gases and vapors varies with different charges, the controlling feature being the substantial removal of gases and vapors. In this specific example, the furnace pressure was 170 microns of mercury. The subsequent evacuation was sufficient to rapidly remove the gases and vapors evolved during the heating and pressing, and in this example, the pressure ranged from 500 to 1000 microns of mercury.

EXAMPLE 11

(Specimen B of tables)

An intermetallic compound-composition powder, in an amount of 254 grams, consisting of 51.6%, by weight, of beryllium, with the balance consisting essentially of niobium, and having a particle size of 200 mesh or finer, was placed in a graphite die in a furnace. Following the procedure of Example 10, the temperature and mechanical pressure were increased to a maximum of about 1520° C. and 2000 p.s.i. concurrently during the maintenance of the vacuum. The mechanical pressure was then discontinued, and the concurrent maximum temperature and vacuum maintained for about a half hour, after which time the heating was discontinued, and the furnace cooled under vacuum to room temperature. The body, upon removal from the furnace, weighed 217.5 grams, and had a density of 2.99 g./cc., which is 99.7% of the calculated theoretical density. Upon chemical analysis, its beryllium content was found to be 51.6%, by weight, of the body, with the balance consisting essentially of niobium, and its grain size was 15 microns.

The furnace pressure during the initial evacuation was 500 microns of mercury, and during the subsequent evacuation ranged from 300 to 500 microns of mercury.

EXAMPLE 12

(Specimen E of tables)

Following the procedure of Example 10, 309 grams of powdered intermetallic compound-composition consising of 46.4% by weight of beryllium, and the balance consisting essentially of niobium, and having a particle size of 200 mesh or finer, were placed in the die. During the concurrent maintenance of temperature, mechanical pressure, and vacuum, the gradual increase was to a maximum temperature of about 1520° C. and to a mechanical pressure of about 2000 p.s.i. The pressure was then discontinued and the concurrent maximum temperature and evacuation were continued for about a half hour, after which the heating was discontinued and the furnace cooled under vacuum to room temperature. The body, upon removal from the furnace, weighed 297 grams and had a density of 3.08 g./cc., which is 97.5% of the calculated theoretical density. The grain size was 15 microns and chemical analysis showed the body to contain beryllium in an amount, by weight of 46.4% of the body, with the balance consisting essentially of niobium.

The pressure during the initial substantial evacuation was 200 microns of mercury, and during the subsequent evacuation ranged from 180 to 250 microns of mercury.

EXAMPLE 13

(Specimen F of tables)

Following the procedure of Example 10, 264 grams of an intermetallic compound-composition consisting of 45.2%, by weight, of beryllium and the balance consisting essentially of niobium, in powder form, and having a particle size of 200 mesh or finer, were introduced into a die. The increase in temperature and pressure were to a maximum temperature of about 1550° C. and a maximum mechanical pressure of about 2000 p.s.i., the pressure, vacuum, and temperature application being maintained concurrently. When the maximum temperature was reached, the pressure was discontinued, and concurrent evacuation and the maximum temperature were maintained for about a half hour. The heating was then discontinued, and the furnace cooled under vacuum to room temperature. Upon removal from the furnace, the body weighed 235 grams, and had a density of 99.4% of the calculated theoretical density, with a grain size of 9 microns. Chemical analysis showed it to contain beryllium in an amount, by weight, of 45.2% of the body, with the balance consisting essentially of niobium.

The pressure during the initial substantial evacuation was 40 microns of mercury, and during the subsequent evacuation, ranged from 100 to 250 microns of mercury.

EXAMPLE 14

(Specimen G of tables)

An intermetallic compound-composition, in powdered form and in the amount of 163.2 grams, consisting of 56% by weight of beryllium and the balance consisting essentially of zirconium, and having a particle size of 200 mesh or finer, was introduced into the die within a furnace. Following the procedure of Example 10, the furnace was evacuated concurrently with the application of about 1000 p.s.i. of mechanical pressure. The vacuum was maintained and heat was applied, accompanied by a concurrent increase of mechanical pressure. During the concurrent evacuation and application of mechanical pressure and heat, a maximum temperature of about 1550° C. and a maximum pressure of about 2000 p.s.i. were attained. At this time, the pressure was discontinued, and the evacuation and temperature were maintained concurrently for about a half hour, after which time the furnace and die were cooled under vacuum to room temperature. The body, upon removal from the furnace, weighed 154.9 grams, and had a density of 2.77 g./cc., which is 99.3% of the calculated theoretical density. The grain size was 25 microns. The body was found by chemical analysis to contain beryllium is an amount equal to 56% of the body, with the balance substantially only zirconium.

The pressure during the initial substantial evacuation was 180 microns of mercury, and during the subsequent evacuation, was about 1000 microns of mercury.

EXAMPLE 15

(Specimen I of tables)

In accordance with the procedure of Example 10, 513 grams of an intermetallic compound-composition in powder form, consisting of 51.5% by weight of beryllium and the balance consisting essentially of zirconium, and having a particle size of 200 mesh or finer, were introduced into the die in a furnace. During the concurrent application of heat, mechanical pressure and evacuation, the increase of temperature was up to a maximum temperature of about 1550° C. The increase in mechanical pressure was up to a maximum of about 2000 p.s.i. The maximum pressure was then discontinued, and the maximum temperature, with evacuation, maintained for about 40 minutes. The heating was then discontinued, and the furnace allowed to cool to room temperature, under vacuum. The body, upon removal, weighed 488.6 grams, and had a density of 2.84 g./cc., corresponding to 98.6% of the calculated theoretical density. The grain size was 24 microns. Chemical analysis disclosed 51.5% of beryllium by weight, of the body, and the balance consisting essentially of zirconium.

The pressure during the initial substantial evacuation was 150 microns of mercury, and during the subsequent evacuation ranged from 75 to 150 microns of mercury.

EXAMPLE 16

(Specimen M of tables)

Following the procedure of Example 10, 240 grams of

an intermetallic compound-composition powder, consisting of 45.7% by weight of beryllium, the balance consisting essentially of zirconium, and having a particle size of 200 mesh or finer, were introduced into the die. During the evacuation with a concurrent increase in mechanical pressure and in temperature, a maximum temperature of about 1550° C. and a maximum mechanical pressure of about 2000 p.s.i. were attained. The mechanical pressure was then discontinued, and the concurrent evacuation and maximum temperature maintained for about a half hour, after which time the heating was discontinued and the furnace vacuum cooled to room temperature. The body, upon removal, weighed 225 grams, and had a density of 3.06 g./cc., corresponding to 100% of the calculated theoretical density. The grain size was 30 microns, and chemical analysis showed the body to contain beryllium in an amount 45.7% by weight, of the body, the balance consisting essentially of zirconium.

The pressure during the initial substantial evacuation was 40 microns of mercury, and during the subsequent evacuation ranged from 200 to 500 microns of mercury.

EXAMPLE 17

(Specimen N of tables)

In accordance with the procedure of Example 10, an intermetallic compound-composition in powder form, in an amount of 345 grams, and consisting of 37.2% by weight of beryllium and the balance consisting essentially of tantalum, and having a particle size of 200 mesh or finer, was introduced into the die, and the furnace was then evacuated while applying about 1000 p.s.i. of mechanical pressure. When the furnace was substantially evacuated, heat was applied and increased, accompanied by a concurrent increase in the mechanical pressure to about 1550° C. and about 2000 p.s.i. Thereupon, the pressure was discontinued, and the maximum temperature and concurrent evacuation was maintained for about a half hour, after which time the heating was discontinued, and the furnace vacuum cooled to room temperature. The body, when removed, weighed 328 grams and had a density of 4.11 g./cc., corresponding to 96.9% of the calculated theoretical density. The grain size was 12 microns, and chemical analysis showed 37.2% by weight of beryllium with the balance consisting essentially of tantalum.

The pressure during the initial substantial evacuation was 800 microns of mercury, and during the subsequent evacuation was about 800 microns of mercury.

EXAMPLE 18

(Specimen Q of tables)

In accordance with the procedure of Example 10, 900 grams of an intermetallic compound-composition consisting of 29.8% by weight of beryllium and the balance consisting essentially of tantalum, and having a particle size of 200 mesh or finer is disposed in the graphite die in a furnace. During the concurrent evacuation, and application of increasing heat and pressure, the maximum temperature attained was about 1550° C. and maximum mechanical pressure of about 2000 p.s.i. When these maximums were reached, the mechanical pressure was discontinued and the evacuation and maximum temperature continued for about a half hour. The heating was then discontinued, the vacuum maintained, and the furnace and die cooled to room temperature. The body, when removed, weighed 855.5 grams and had a density of 4.88 g./cc., corresponding to 96% of the calculated theoretical density. The grain size was 18 microns. Chemical analysis showed 29.8% of beryllium, by weight of the body, the balance consisting essentially of tantalum.

The pressure during the initial substantial evacuation was 200 microns of mercury, and during the subsequent evacuation ranged from 175 to 250 microns of mercury.

EXAMPLE 19

(Specimen R of tables)

In accordance with the procedure of Example 10, 250 grams of an intermetallic compound-composition consisting of 53% by weight of beryllium and the balance consisting essentially of molybdenum, and having a particle size of 200 mesh or finer, were introduced in the die in a furnace. During the concurrent evacuation and the increase in application of mechanical pressure and heat, a maximum temperature of about 1550° C. and a maximum pressure of about 2000 p.s.i. were reached. As soon as this maximum temperature was reached, the pressure was discontinued, and the concurrent maximum temperature and the evacuation were continued for about a half hour. The heating was then discontinued, the vacuum continued until the furnace is cooled to room temperature. The body weighed 236 grams, and had a density of 3.02 g./cc., corresponding to 97.7% of the calculated theoretical density. The grain size was 16 microns and chemical analysis showed 53% beryllium by weight of the body, the balance consisting essentially of molybdenum.

The pressure during the initial substantial evacuation was 45 microns of mercury, and during the subsequent evacuation ranged from 200 to 500 microns of mercury.

EXAMPLE 20

(Specimen S of tables)

An intermetallic compound-composition, in an amount of 205.65 grams, and consisting of 46.5% by weight of aluminum and the balance consisting essentially of niobium, and having a particle size of 200 mesh or finer, was introduced in the graphite die. In accordance with the procedure of Example 10, the furnace was then evacuated at room temperature during a concurrent application of about 1000 p.s.i. mechanical pressure. When the furnace was substantially evacuated, heat was applied and the pressure was concurrently gradually increased, the evacuation being continued. The concurrent evacuation, application of heat, and increase in mechanical pressure continued until a maximum temperature of about 1465° C. and a maximum pressure of about 2000 p.s.i. were reached. The pressure was then discontinued, and the concurrent maximum temperature and evacuation maintained for about a half hour, after which the heating was discontinued, the vacuum continued and the furnace cooled to room temperature. The body was then removed from the furnace. It weighed 197.5 grams, and had a density of 4.36 g./cc., which corresponds to 95.4% of the calculated theoretical density. The grain size was 25 microns and chemical analysis showed 46.5% aluminum by weight of the body, with the balance consisting essentially of niobium.

EXAMPLE 21

(Specimen T of tables)

Following the procedure of Example 10, 346 grams of an intermetallic compound-composition consisting of 30.9% by weight of aluminium and the balance consisting essentially of tantalum, and having a particle size of 200 mesh or finer, were introduced into a graphite die located in a furnace. During the concurrent evacuation and increase of heat and mechanical pressure, a maximum temperature of about 1465° C. and a maximum pressure of about 2000 p.s.i. were reached. As soon as the maximum temperature and pressure were reached, the mechanical pressure was discontinued, and the maximum temperature and evacuation maintained for about a half hour. With the evacuation continuing, the furnace was cooled to room temperature. The body was then removed, and weighed 288.8 grams, and had a density of 6.60 g./cc., which corresponds to 95.4% of the calculated theoretical density. Chemical analysis showed 30.9% aluminum, by weight of the body, with the balance consisting essentially of tantalum.

Table I

TRANSVERSE-RUPTURE STRENGTH DATA FOR INTERMETALLIC COMPOUND-COMPOSITIONS

Specimen	Nominal Composition [a]	Test Temp. (° F.)	Modulus of Rupture (p.s.i.)	Young's Modulus (10^6 p.s.i.)
A—Example 10	53.6% Be, 46.4% Nb	2,300	39,300	40
		2,500	39,300	40
		2,750	18,700	20
B—Example 11	51.6% Be, 48.4% Nb	2,300	55,800	24
		2,500	53,500	18
		2,750	[b] 32,000	8
C	50.2% Be, 49.8% Nb	2,300	49,900	17
		2,500	50,000	11
		2,750	[b] 28,800	4
D	48.9% Be, 51.1% Nb	2,300	71,100	13
		2,500	63,400	7
		2,750	[b] 36,900	3
E—Example 12	46.4% Be, 53.6% Nb	2,300	71,100	14
		2,500	63,200	10
		2,750	[b] 35,200	4
F—Example 13	45.2% Be, 54.8% Nb	2,300	39,000	22
		2,500	[b] 37,500	20
		2,750	[b] 16,400	----
G—Example 14	56.0% Be, 44.0% Zr	2,300	36,900	14
		2,500	37,400	15
		2,750	24,600	10
H	54.1% Be, 45.9% Zr	2,300	35,200	20
		2,500	44,500	24
		2,750	33,600	16
I—Example 15	51.5% Be, 48.5% Zr	2,300	43,600	25
		2,500	[b] 56,400	15
		2,750	[b] 26,700	15
J	49.8% Be, 50.2% Zr	2,300	31,800	----
		2,500	32,700	----
		2,750	36,600	----
K	48.3% Be, 51.7% Zr	2,300	35,900	21
		2,500	51,000	21
		2,750	[b] 25,200	4
L	47.1% Be, 52.9% Zr	2,500	38,090	17
		2,750	31,700	----
M—Example 16	45.7% Be, 54.3% Zr	2,300	39,600	25
		2,500	27,200	15
		2,750	24,400	10
N—Example 17	37.2% Be, 62.8% Ta	2,300	53,200	24
		2,500	36,000	14
		2,750	26,000	10
O	35.5% Be, 64.5% Ta	2,300	47,000	26
		2,500	37,800	12
		2,750	[b] 18,700	10
P	32.0% Be, 68.0% Ta	2,300	56,800	20
		2,500	[b] 37,500	19
		2,750	[b] 18,700	15
Q—Example 18	29.8% Be, 70.2% Ta	2,300	66,300	15
		2,500	53,500	11
		2,750	30,600	10
R—Example 19	53.0% Be, 47.0% Mo	2,300	41,800	15
		2,500	29,900	12
		2,750	11,800	1
S—Example 20	46.5% Al, 53.5% Nb	2,300	19,600	10
		2,300	20,800	6
T—Example 21	30.9% Al, 69.1% Ta	2,300	18,100	13
		2,300	19,100	15

[a] Approximate composition disregarding minor amounts of impurities.
[b] Specimens deflected to the limit allowable in the test apparatus, but did not rupture. Modulus of rupture reported is based on the loading of the specimen at the moment deflection ceased.

Table II

OXIDATION TEST DATA FOR INTERMETALLIC COMPOUND-COMPOSITIONS

Specimen	Nominal Composition [a]	Specimen Density (Percent of theoret.)	Test Temp. (° F.)	Atmosphere [b]	Weight Gain 100 hr. (mg./cm.^{2})	Mils Penetration
A—Ex. 10	53.6% Be, 46.4% Nb	99.0	2,500	Dry air	10.7	1.3
		98.7	2,500	Moist air	5.1	0.6
B—Ex. 11	51.6% Be, 48.4% Nb	100	2,500	Dry air	3.6	0.4
		99.4	2,700	Moist air	17.3	2.0
F—Ex. 13	45.2% Be, 54.8% Nb	98.8	2,700	Dry air	17.4	2.0
		97.3	2,700	Moist air	17.3	2.0
G—Ex.14	56.0% Be, 44.0% Zr	100	2,800	Dry air	10.8	1.4
		95.4	2,900	Ambient air	22.8	2.9
		99.0	2,500	Moist air	6.1	0.8
H	54.1% Be, 45.9% Zr	100	2,600	Dry air	7.8	1.0
M—Ex. 16	45.7% Be, 54.3% Zr	99.5	2,700	Dry air	9.4	1.2
N—Ex. 17	37.2% Be, 62.8% Ta	100	2,800	Dry air	9.2	1.1
		100	2,500	Moist air	3.5	0.4
O	35.5% Be, 64.5% Ta	100	2,500	Dry air	5.9	0.7
		96.5	2,700	Dry air	7.5	0.9
Q—Ex. 18	29.8% Be, 70.2% Ta	97.7	2,300	Dry air	2.0	0.2
		100	2,300	Moist air	6.0	0.7
		100	2,800	Dry air	29.0	3.2
R—Ex. 19	53.0% Be, 47.0% Mo	98.0	2,700	Dry air	6.8	0.6
		98.7	2,500	Moist air	9.7	0.8
S—Ex. 20	46.5% Al, 53.5% Nb	92.0	2,500	Dry air	10.2	1.3
		99.3	2,500	Moist air	7.8	1.0
T—Ex. 21	30.9% Al, 69.1% Ta	98.8	2,600	Dry air	6.9	0.9
		99.4	2,500	Moist air	5.0	0.6

[a] Approximate composition disregarding minor amounts of impurities.
[b] Dry air=less than 0.1 mg. of water per liter (effluent gas at room temperature).
Moist air=12 mg. of water per liter (effluent gas at room temperature).

This application is a division of our pending application Serial No. 27,523, filed May 9, 1960, titled Intermetallic Compound-Composition and Bodies Composed Thereof and Method of Making the Same, which is directed to certain bodies, the present application being directed to the method of making same.

Having thus described our invention, we claim:

1. The method of making fine-grained high density bodies of predetermined size and shape, which bodies, at temperatures of from 2000 to 2900° F., have high strength and high oxidation resistance, and comprising placing in a die a powder, of particle size of 200 mesh or finer, consisting of one metal selected from a first group consisting of beryllium and aluminum, and one metal M selected from a second group consisting of niobium, zirconium, molybedenum and tantalum, the metal of the first group being, by weight, not more than a predetermined amount, and the balance consisting essentially of the metal M, said predetermined amount being, for beryllium, not to exceed substantially stoichiometric proportions corresponding to MBe_{13}, and for aluminum not to exceed substantially the stoichiometric proportion corresponding to MAl_3, then subjecting the powder in the die to a vacuum and concurrently therewith applying and maintaining on the powder in the die mechanical pressure at about 1000 pounds per square inch until the air and vapors are substantially removed therefrom, then, while maintaining the vacuum and mechanical pressure, gradually heating the powder in the die and, before sintering temperature of the powder is reached, increasing the pressure until a higher mechanical pressure sufficient to result in compaction of the sintered material to substantially theoretical density and said sintering temperature are reached, then discontinuing the application of the mechanical pressure and maintaining the said sintering temperature for a period sufficient to anneal the body, and then allowing the body to cool slowly to room temperature.

2. A method according to claim 1 wherein said sintering temperature is from about 1350° C. to 1650° C.

3. A method according to claim 1 wherein said higher mechanical pressure is about 2000 pounds per square inch.

4. A method according to claim 1 wherein said sintering temperature is from about 1350° C. to 1650° C., and said higher mechanical pressure is about 2000 pounds per square inch.

5. A method according to claim 1 wherein the amount of the metal of the first group is at least about 29.8%, by weight, of the weight of the body.

6. The method according to claim 1 wherein said vacuum is maintained for said period during which said sintering temperature is maintained following the discontinuance of the application of the mechanical pressure.

7. The method according to claim 6 wherein said vacuum is maintained also during the cooling of the body to room temperature.

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